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(54) Title: ANTISTATIC PEROXIDE COMPOSITION		
(57) Abstract		
<p>The invention pertains to a composition comprising a dialkyl peroxide and an effective amount of an antistatic. The dialkyl peroxide preferably is di-tert-butyl peroxide or di-tert-amyl peroxide. The antistatic is an organic acid or a salt thereof or a salt of an inorganic acid or a mixture of these substances. The antistatic may be a single substance, a polymeric substance, or a composition or mixture of substances. The invention further pertains to the use of said composition, int. al., (co)-polymerisation reactions.</p>		

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Antistatic peroxide composition

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The invention pertains to a novel peroxide composition and the use of same.

10 A major problem in the preparation, storage, transport, and use of peroxide compositions is the occurrence of static electricity. Discharge of this static electricity creates the hazard of electric shocks and sparks. Electric shocks are annoying and can on occasion be hazardous to man. When the atmosphere is inflammable, sparks can lead to fires and/or explosions, e.g., because of the volatility of the employed peroxide.

15

In general, there is a risk of static electricity building up when the electric conductivity of a peroxide composition is low. For instance, di-tert-amyl peroxide (Trigonox[®] 201) has an electric conductivity of less than 20 pS/m. There is the risk of a particularly hazardous situation when both the electric conductivity and the flash point of a peroxide composition are low. This is what happens, e.g., in the case of di-tert-butyl peroxide (Trigonox[®] B), which has an electric conductivity of 0,4 pS/m and a flash point of 6°C. By contrast, the flash point of di-tert-amyl peroxide is 29°C. As a rule, it holds that when a peroxide composition has an electric conductivity of less than 50 pS/m and a flash point below 40°C, there is question of a hazardous situation such as described above. The use of dilutions in non-conductive solvents, such as hydrocarbons, can also cause a low(er) electric conductivity of peroxide compositions. For example, Trigonox[®] B-C30, a 30% solution of di-tert-butyl peroxide in isododecane, still has a dangerously low electric conductivity and flash point.

20 25 30 The continuous phase of water-in-oil emulsions, in which the continuous phase comprises a dialkyl peroxide, will also have a low electric conductivity.

A solution has now been found to the problem of peroxide compositions having a low electric conductivity.

The peroxide composition according to the present invention is characterised in that the composition comprises a dialkyl peroxide and an effective amount of an antistatic, with the proviso that the composition is not an oil-in-water emulsion.

It should be noted that JP-A-63203660 discloses an oil-in-water emulsion comprising 50% di-tert-butyl peroxide, 0,5% acetic acid, 0,5% sodium dodecylbenzene sulfonate, and 49% water, in which sodium dodecylbenzene sulfonate is used as the emulgator. Such oil-in-water emulsions wherein water is the continuous phase do not have the problem of a low conductivity.

One major advantage of the antistatic peroxide composition according to the invention consists in that there is no build-up of charge during transport in pipes, stirring, and other process treatments, and that it can be packed safely in the usual non-conductive containers for transport and storage. Also, the conductivity of the peroxide composition is high enough during the required storage period, and because only a small amount of antistatic has to be added to give the composition sufficient electric conductivity, there are no interfering influences when the composition is used.

The dialkyl peroxide composition according to the invention preferably comprises a di-tert-alkyl peroxide. Most preferred is a composition comprising di-tert-butyl peroxide (Trigonox® B) or di-tert-amyl peroxide (Trigonox® 201).

Preferably, the peroxide composition contains 5-99,9 per cent by weight of this peroxide, calculated on the total weight of the composition. Typical examples include commercially available grades of dialkyl peroxides, such as 90%

Trigonox[®] 201, 99% Trigonox[®] B and a 30 per cent by weight solution of the latter dialkyl peroxide in isododecane, i.e., Trigonox[®] B-C30.

The term antistatic in the present description refers not only to single substances but also to polymers and compositions and mixtures of these substances. By an effective amount of an antistatic is meant an amount which will give a peroxide composition having an electric conductivity exceeding 50 pS/m. Preferably, the peroxide composition according to the invention comprises less than 1000 ppm (i.e. less than 0,1 per cent by weight) of an antistatic, based on the weight of dialkyl peroxide in the composition. For that matter, the electric conductivity is determined with the aid of an Eltex Tera Ohm meter and a conductivity cell for liquids, as described in the experimental section. Most preferred is a peroxide composition comprising less than 400 ppm of an antistatic, which amount results in an electric conductivity exceeding 1000 pS/m. The peroxide composition according to the present invention usually contains 0,5-50 ppm of an antistatic.

Good electric conductivity of the peroxide composition according to the present invention is obtained when the antistatic comprises an organic acid or a salt thereof, a salt of an inorganic acid, or a mixture of one or more of these substances.

Suitable organic acids are polymeric compounds containing one or more sulphuric acid groups, sulphonic acid groups, carboxylic acid groups, phosphoric acid groups or phosphonic acid groups. Other suitable organic acids are sulphuric acids, sulphonic acids, carboxylic acids, phosphoric acids or phosphonic acids or hydrates thereof substituted with one or more C₁-C₂₀ alkyl radicals, C₂-C₂₀ alkenyl radicals, C₆-C₁₈ aryl radicals, C₇-C₃₀ aralkyl radicals or C₇-C₃₀ alkaryl radicals, said radicals being linear or branched and containing one

or more hydroxy groups, alkoxy groups, carboxy groups, amide groups or ester groups or not.

Suitable salts of organic acids are ammonium and quaternary ammonium sulphates, ammonium and quaternary ammonium sulphonates, ammonium and quaternary ammonium carboxylates, ammonium and quaternary ammonium phosphates or ammonium and quaternary ammonium phosphonates or hydrates thereof substituted in the anion or the cation with one or more C₁-C₂₀ alkyl radicals, C₂-C₂₀ alkenyl radicals, C₆-C₁₈ aryl radicals, C₇-C₃₀ aralkyl radicals or C₇-C₃₀ alkaryl radicals, said radicals being linear or branched and containing one or more hydroxy groups, alkoxy groups, carboxy groups, amide groups or ester groups or not. Metal ion salts of said anions are also suitable for use. Examples of suitable metal ions are alkali metal ions, alkaline-earth metal ions or transition metal ions.

Suitable salts of inorganic acids are ammonium and quaternary ammonium halides substituted with one or more C₁-C₂₀ alkyl radicals, C₂-C₂₀ alkenyl radicals, C₆-C₁₈ aryl radicals, C₇-C₃₀ aralkyl radicals or C₇-C₃₀ alkaryl radicals, said radicals being linear or branched and containing one or more hydroxy groups, alkoxy groups, carboxy groups, amide groups or ester groups or not. The corresponding sulphates or phosphates are suitable for use also.

To the skilled person it will be a simple matter to select an appropriate antistatic from these acids and salts, depending on the peroxide to be used and the desired application. Preferably, the antistatic will comprise one or more of the following substances: para-toluene sulphonic acid monohydrate, dodecyl benzene sulphonic acid, ethoxylated and phosphatised nonyl phenol, copper or chromium dialkyl salicylate, sodium (sec-C₁₃-C₁₇-alkyl)sulphonate, sodium or calcium dialkyl sulphosuccinate, di(hydrogenated tallow)-dimethyl ammonium methosulphate, didecyl dimethyl ammonium chloride, a polymeric compound

containing sulphonic acid groups, sulphonate groups, and sulphone groups or a polymeric compound containing carboxylic acid groups and amide groups. An example of a suitable antistatic is ASA-3.

- 5 If so desired, the peroxide composition according to the invention may comprise a solvent, diluent or oil, or a mixture of these. Liquids known to the skilled person are alcohols, e.g., n-butyl alcohol, tert-butyl alcohol, tert-amyl alcohol, n-propyl alcohol, and isopropyl alcohol; hydrocarbons, e.g., isododecane; esters of dicarboxylic acids, e.g., dibutyl phthalate; and silicone oils, white oils, and mineral
10 oils. It should be noted in this connection that some liquids may not be suitable for use in the peroxide composition of the invention, for instance because said liquids will create problems when the peroxide composition is applied. It should further be noted that generally the presence of a solvent, diluent or oil increases the electric conductivity of the composition and, hence, reduces the occurrence
15 of static electricity and its dangers. This, however, is not the case when hydrocarbons such as isododecane are used as discussed above.

The peroxide composition of the invention can also be a water-in-oil emulsion wherein the dialkyl peroxide is the continuous phase.

- 20 Preferably, the antistatic is dissolved in an appropriate medium, such as mentioned earlier, before it is added to the peroxide or a formulation thereof. In this case it is important to ensure that the antistatic is in solution at the temperature at which the peroxide composition is stored, shipped, and handled. Preferably, less than 0,5 per cent by weight of the medium, calculated on the
25 peroxide weight, will be present in the peroxide composition.

- Summarizing, the antistatic composition according to the present invention comprising a dialkyl peroxide can be a single phase composition, such as the substantially pure dialkyl peroxide or a solution of the dialkyl peroxide, e.g., in
30 isododecane, or it can be a composition comprising a continuous phase and a

discontinuous phase, such as a water-in-oil emulsion, wherein the continuous phase comprises the dialkyl peroxide.

5 The antistatic dialkyl peroxide composition of the present invention can be used as a radical initiator in (co)polymerisations, curing and cross-linking of rubbers and thermosetting formulations, and can be used in (co)polymer modifications and chemical reactions involving peroxides.

10 The invention will be illustrated in greater detail with reference to the following, unlimitative examples.

EXPERIMENTAL SECTION

Materials

15	Tx B	Trigonox® B, di-tert-butyl peroxide, ex Akzo Nobel
	DTAP	Trigonox® 201, di-tert-amyl peroxide, ex Akzo Nobel
	Aerosol OT-100	Sodium di(2-ethylhexyl)sulphosuccinate, 100%, ex Cytec
	Armac C	Cocoamine acetate, ex Akzo Nobel
20	Armostat 800	Triethanolamine n-dodecyl benzene sulphonate, 53% in water, ex Akzo Nobel
	Arquad 2.10	Didecyl dimethyl ammonium chloride, 83%, ex Akzo Nobel
	Arquad 2T-70	Di-tallow-dimethyl ammonium chloride, 70% in 2-propanol, ex Akzo Nobel
25	Arquad M2HT-90MS	Di-(hydrogenated tallow)-dimethyl ammonium metho-sulphate, ex Akzo Nobel
	ASA-3	A mixture of 20% chromium(III) di(C ₁₆ -C ₁₈ -alkyl)-salicylate, 10% calcium bis(diisodecyl sulphosuccinate), 45% 2-

		methyl-5-vinyl pyridine (C ₁₆ -C ₁₈ -alkyl) methacrylate copolymer, 25% <i>meta</i> -xylene, ex Shell
	BSA	Benzene sulphonic acid, 90%, ex Acros
	CuDIPS	Cu(II)3,5-diisopropylsalicylate hydrate, ex Aldrich
5	DBSA	Dodecyl benzene sulphonic acid, 97%, ex Janssen
	Gafac RM710	anionic soap, 100%, ex GAF Chemical Corp.
	Marlon PS65	Sodium (sec-C ₁₃ -C ₁₇ -alkyl)sulphonate, 65%, ex Hüls
	Marlon-NH4	Ammonium salt of Marlon PS65
	MSA	Methyl sulphonic acid, >99%, ex Aldrich
10	PAPSA	Poly(2-acrylamido-2-methyl-1-propane sulphonic acid), dried, ex Aldrich
	PEMA	Copolymer of methylvinyl ether and maleic anhydride, Mw 311000, ex Aldrich
15	Servon XCA 850	Calcium bis(di-isodecyl sulphosuccinate), 50% in xylene, ex Hüls
	Servoxyl VPI 55	Phosphoric acid, butyl ester, sodium salt, 50-100%, ex Hüls
	Servoxyl VPIZ 100	1-butanol, phosphatised, 50-100%, ex Hüls
20	Servoxyl VPNZ 5/100	(or NPEP) Nonyl phenol, ethoxylated, phosphatised, ex Hüls
	Stadis 450	A mixture of, int. al., DBSA, a quaternary ammonium salt, and a polymeric compound containing sulphonic acid groups, sulphonate groups, and sulphone groups, in toluene-isopropylalcohol, ex Du Pont (Octel)
25	TBAI	Tert-butyl alcohol, ex Baker
	TBA _m	Tributyl amine, ex Baker
	TEP	Triethyl phosphate, ex Baker
	TSA	Toluene-4-sulphonic acid.H ₂ O, > 99%, ex BDH AnalaR
	TIM	Toluene-isopropylalcohol-methanol mixture
30	ZFA	Zirconium(IV)trifluoroacetylacetonate, 97%, ex Aldrich

Electric conductivity

The electric conductivity, expressed in pS/m, was measured with the aid of an Eltex Tera Ohm Meter (type 6206) and a stainless steel immersion cell consisting of two concentrically arranged cylindrical electrodes, with the cell constant being $K=1,65 \text{ 1/m}$. The conductivity was measured one day after the samples were prepared. The measurements were carried out at room temperature (19-23°C). The resistivity was read off one minute after the immersion of the cell.

- 10 In Table I the results obtained with di-tert-butyl peroxide (Trigonox® B, Tx B) are given for Comparative examples A-D and Examples 1-16. The amount of antistatic is expressed in mg per kg (ppm) Tx B.

Table I
Conductivity of Tx B containing antistatic

	Antistatic	Concentration (mg/kg)	Conductivity (pS/m)
A	None	0	0,4
B	TSA	51	3
C	TBAI	1500	2,5
D	Arquad M2HT-90MS, 13% in TBAI	250	3,5
1	DBSA	59	60
2	Gafac RM710, 30 min. ultrasonic	110	70
3	Gafac RM710, 10% in TBAI	1000	120
4	Armostat 800	1000	750
5	Armac C	50	50
6	Aerosol OT-100	50	165
7	Aerosol OT-100	260	525
8	Marlon PS65, 10 min. ultrasonic	99	750
9	Marlon PS65, 10 min. ultrasonic	145	2750
10	Marlon-NH4	150	2000
11	CuDIPS	251	1100
12	ZFA	253	600
13	Stadis 450	5	750
14	Servoxyl VPNZ 5/100	50	1050
15	Servoxyl VPNZ 5/100	249	7000
16	ASA-3	4.7	2360
17	PAPSA	49	61
18	Servon XCA 850	255	500

Marlon-NH₄ was prepared by passing a solution of Marlon PS65 in water over a highly acidic ion exchanger and neutralising the eluate with 1% of NH₄OH to pH 6. The resulting salt was obtained by evaporation of the water in vacuum.

- 5 Table I shows that the use of Aerosol OT-100, Marlon PS65, CuDIPS, ZFA, Stadis 450, Servoxyl VPNZ 5/100, ASA-3, and Servon XCA 850 in particular can lead to a substantial improvement of the electric conductivity of Tx B.

Stability

- 10 Table II shows the data of a test of the stability of Tx B compositions containing 247 ppm of Marlon PS65.

Table II

Conductivity of Tx B containing Marlon PS65

15

Storage time (weeks)	Conductivity (pS/m) after storage at:	
	-10°C	40°C
0,15	6600	
0,6	4850	8550
1	3950	7500
2,8	3300	4700
5,15	3000	5800
14,7	2750	3235

It is clear from Table II that while the conductivity of Tx B containing 247 ppm of Marlon PS65 slowly decreases during storage, it remains high enough to prevent static charging of the peroxide in properly earthed systems.

Table III shows the data for Comparative examples E and F and Examples 19-29. First, a 15-20 wt.% solution of an acid and an amine or a quaternary amine was prepared, which was then added to Tx B. The concentration represents the amount of antistatic solution in mg per kg (ppm) of Tx B. The molar ratio indicates the ratio between the acid and the Arquad or the amine.

Table III
Conductivity of Tx B containing antistatic

	Antistat.	Conc. (mg/kg)	Acid	Arquad / amine	Solvent	Molar ratio	Conduct. (pS/m)
E	Astat2	1026	DBSA	Arq. 2T-70	TIM	1:0,62	15
F	Astat 3	999	DBSA	TBAm	TIM	1:0,52	15
19	Astat5	966	DBSA	Arq.2.10	TIM	1:0,43	65
20	Astat6	500	TSA	Arq.M2HT-90MS	TBAI	1:0,50	5050
21	Astat6	250	TSA	Arq.M2HT-90MS	TBAI	1:0,50	1550
22	Astat6	50	TSA	Arq.M2HT-90MS	TBAI	1:0,50	75
23	Astat7	1008	MSA	Arq.M2HT-90MS	TBAI	1:0,504	3400
24	Astat9	1008	TSA	TBAm	TBAI	1:1,03	85
25	Astat18	250	BSA	Arq.M2HT-90MS	TBA/IPA	1:0,50	310
26	Astat 20	77		1)		1:1,53	440
27	Astat 21	210		2)		1:0,50	85
28	Astat 23	250		3)		1:0,80	780
29	Astat 46	294		4)		1:2	190

1) Astat 20 is composed of a mixture of CuDIPS and Aerosol OT-100 in a molar ratio of 1:1,53.

2) Astat 21 is composed of a mixture of a solution in TBAI of Arq.M2HT-90MS and Aerosol OT-100 in a molar ratio of 1:0,50.

5 3) Astat 23 is composed of a mixture of a solution in TBAI of Arq.M2HT-90MS and Servoxyl VPNZ 5/100 in a molar ratio of 1:0,80.

4) Astat 46 is the reaction product of PEMA and dodecyl amine, 17,6% in xylene in a molar ratio of 1:2.

10 From Table III it can be deduced that mixture Astat6, which is a solution of Arq.M2HT-90MS and TSA in TBAI, provides a substantial improvement of the electric conductivity of Tx B.

15 The applicational and safety testing of Tx B was performed with the aid of a control sample Tx B and a sample to which an amount of 1000 mg of antistatic per kg of Tx B was added. The antistatic consisted of: 69 mg of para-toluene sulphonic acid monohydrate, 122 mg of Arquad M2HT-90MS, and 810 mg of tert-butyl alcohol as solvent.

20 Use of Tx B in the preparation of polyacrylate

The effect of an antistatic from Tx B on the acrylate properties was determined by means of a model reaction performed with a monomer mixture in a solvent. The monomer mixture was composed of 40% butyl acrylate, 20% styrene, 28% hydroxyethyl methacrylate, 10% methyl methacrylate, and 2% methacrylic acid.

25 The reaction was carried out at 165°C in Solvesso 100, the initiator was added over a period of 4 hours.

The solids content was determined as the percentage of non-volatile material after 30 minutes at 150°C. The molecular weight was determined by HPLC analysis,

with standard polystyrene samples being used as controls. The colour was determined in a "Lovibond" APHA meter. The results are listed in Table IV.

Table IV

5 Results when using Tx B in high-solids acrylate preparation

System	Solids (%)	Mw	Mn	Colour APHA
with antistatic	72,6	4200	2000	30
control	72,9	4100	2000	30

10 From the measuring data of the model system it can be concluded that the antistatic does not have any effect on the polymer properties; the molecular weight, colour, and percentage of solids are comparable.

Safety

15 The safety properties of Tx B containing antistatic were tested in accordance with the UN flow chart. The methods are described in "Recommendations on Transport of Dangerous Goods," Second edition. The results of the UN tests with conductive Tx B are the same as those obtained with a control sample.

20 The product is:

- insensitive to detonation,
- insensitive to deflagration,
- the effect of pressure when heating under specified conditions is "Low" (tests using 50 g were not performed),

- Additional tests:
- the explosive force measured in the HPA (High Pressure Autoclave) is "Not Low,"
 - the SADT is greater than or equal to 90°C
 - the product is insensitive to impact and friction,
 - the electric conductivity exceeds 3000 pS/m, the control sample's conductivity is 0,4 pS/m.

Table V lists the data for di-tert-amyl peroxide (DTAP, Trigonox® 201) in Comparative example G and Examples 30-33. The composition of Astat 6 is to be found in Table III.

Table V
Conductivity of DTAP containing antistatic

	Antistatic	Concentration (mg/kg)	Conductivity (pS/m)
G	None	0	20
30	Marlon PS65	257	900
31	Stadis 450	57	10000
32	Servoxyl VNPZ 5/100	358	4000
33	Astat 6	698	400

It is clear from Table V that Marlon PS65, Stadis 450, and Servoxyl VNPZ 5/100 increase the electric conductivity of DTAP substantially.

Table VI shows the outcome of a stability test of a number of antistatic DTAP compositions after storage at room temperature.

Table VI

Stability of DTAP compositions

Antistatic	Conductivity (pS/m)	
	after 1 day	after 5 weeks
None	20	15
Marlon PS65, 257ppm	900	800
Astat 6, 698 ppm	400	800
Stadis 450, 57 ppm	10000	5300
Servoxyl VPNZ 5/100, 358 ppm	4000	85

It is clear from Table VI that Marlon PS65, Astat 6, and Stadis 450 are stable and provide a DTAP composition of which the good conductivity is permanent.

The preceding examples serve only to illustrate the present invention. The scope of the invention is determined with reference to the claims below.

Claims

1. A peroxide composition, characterised in that the composition comprises a dialkyl peroxide and an effective amount of an antistatic, with the proviso
5 that the composition is not an oil-in-water emulsion.
2. A peroxide composition according to claim 1, characterised in that the dialkyl peroxide is di-tert-butyl peroxide or di-tert-amyl peroxide.
- 10 3. A peroxide composition according to claim 1 or 2, characterised in that the antistatic comprises an organic acid, a salt thereof, a salt of an inorganic acid, or a mixture of one or more of these substances.
- 15 4. A peroxide composition according to claim 3, characterised in that the organic acid is a polymeric compound containing one or more sulphuric acid groups, sulphononic acid groups, carboxylic acid groups, phosphoric acid groups or phosphonic acid groups.
- 20 5. A peroxide composition according to claim 3, characterised in that the organic acid is a sulphuric acid, sulphononic acid, carboxylic acid, phosphoric acid or phosphonic acid or a hydrate thereof substituted with one or more C_1 - C_{20} alkyl radicals, C_2 - C_{20} alkenyl radicals, C_6 - C_{18} aryl radicals, C_7 - C_{30} aralkyl radicals or C_7 - C_{30} alkaryl radicals, said radicals being linear or branched and optionally containing one or more hydroxy groups, alkoxy
25 groups, carboxy groups, amide groups or ester groups.
- 30 6. A peroxide composition according to claim 3, characterised in that the salt of an organic acid is ammonium or quaternary ammonium sulphate, ammonium or quaternary ammonium sulphonate, ammonium or quaternary ammonium carboxylate, ammonium or quaternary ammonium phosphate or

ammonium or quaternary ammonium phosphonate or a hydrate thereof substituted in the anion or the cation with one or more C₁-C₂₀ alkyl radicals, C₂-C₂₀ alkenyl radicals, C₆-C₁₈ aryl radicals, C₇-C₃₀ aralkyl radicals or C₇-C₃₀ alkaryl radicals, said radicals being linear or branched and optionally
5 containing one or more hydroxy groups, alkoxy groups, carboxy groups, amide groups or ester groups.

7. A peroxide composition according to claim 3, characterised in that the salt of an inorganic acid is an ammonium or quaternary ammonium halide,
10 ammonium or quaternary ammonium sulphate, ammonium or quaternary ammonium phosphate substituted with one or more C₁-C₂₀ alkyl radicals, C₂-C₂₀ alkenyl radicals, C₆-C₁₈ aryl radicals, C₇-C₃₀ aralkyl radicals or C₇-C₃₀ alkaryl radicals, said radicals being linear or branched and optionally containing one or more hydroxy groups, alkoxy groups, carboxy groups,
15 amide groups or ester groups.

8. A peroxide composition according to any one of the preceding claims, characterised in that the composition contains less than 1000 ppm of an antistatic, calculated on the weight of peroxide.

9. A peroxide composition according to any one of the preceding claims, characterised in that the composition also comprises a solvent, a diluent or an oil, or a mixture of these.

- 25 10. Use of an antistatic peroxide composition according to any one of preceding claims 1-9 as a radical initiator for (co)polymerisations, curing and cross-linking rubbers and thermosetting formulations, and in (co)polymer modifications and chemical reactions involving peroxides.

INTERNATIONAL SEARCH REPORT

International Application No

PCT/EP 97/06438

A. CLASSIFICATION OF SUBJECT MATTER

IPC 6 C09K3/16 C07C409/00 C08F4/32

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 C09K C07C C08F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	CHEMICAL ABSTRACTS, vol. 110, no. 18, 1 May 1989 Columbus, Ohio, US; abstract no. 155020, IWAMI, HIDEAKI: "Organic peroxide emulsions for polymerization initiators for vinyl monomers" XP002060592 cited in the application see abstract & JP 63 203 660 A (KAYAKU NOURY CORP., JAPAN)	1,3,5,6, 9,10
A	US 5 334 326 A (BOSTICK JOHN V) 2 August 1994 see the whole document --- -/-	1,3,5,9, 10

☒ Further documents are listed in the continuation of box C.

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Date of the actual completion of the international search

30 March 1998

Date of mailing of the international search report

14/04/1998

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Shade, M

INTERNATIONAL SEARCH REPORT

International Application No
PCT/EP 97/06438

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 5 300 600 A (BOCK LAWRENCE A ET AL) 5 April 1994 see column 1, line 1 - column 5, line 25 ---	1, 3-6, 9, 10
A	DATABASE WPI Section Ch, Week 8751 Derwent Publications Ltd., London, GB; Class A60, AN 87-358750 XP002039673 & JP 62 260 801 A (KANEKAFUCHI CHEM KK) , 13 November 1987 see abstract -----	1, 3, 6, 7, 9, 10

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/EP 97/06438

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 5334326 A	02-08-94	NONE	
US 5300600 A	05-04-94	NONE	

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